

(12) **UK Patent Application** (19) **GB** (11) **2 270 576** (13) **A**

(43) Date of A Publication **16.03.1994**

(21) Application No **9315620.6**

(22) Date of Filing **28.07.1993**

(30) Priority Data

(31) **07944215** (32) **11.09.1992** (33) **US**

(71) Applicant(s)

American Optical Corporation

(Incorporated in USA - Delaware)

**14 Mechanic Street, Southbridge,
Massachusetts 01550, United States of America**

(72) Inventor(s)

Nori Y C Chu

Jon Haglund

(74) Agent and/or Address for Service

Kilburn & Strobe

**30 John Street, LONDON, WC1N 2DD,
United Kingdom**

(51) INT CL⁵

G02C 7/10 1/04

(52) UK CL (Edition M)

G2J JS3A

(56) Documents Cited

GB 2174711 A

US 4289497 A

(58) Field of Search

UK CL (Edition L) G2J JFP JS3A JS3X JS6CX JS6C2

JS6DX

INT CL⁵ G02C

WPI

(54) **Method for producing photochromic plastic lens**

(57) A photochromic property is imparted to a plastic lens at high temperatures by microwave heating the lens in a solution of a photochromic dye or in contact with a plastic film containing the dye.

GB 2 270 576 A

METHOD FOR PRODUCING PHOTOCHROMIC PLASTIC LENS

The present invention relates to photochromic plastic lenses. More particularly, the present invention relates to
5 a fast and efficient method for producing photochromic lenses by microwave heating.

Photochromic materials have many potential applications in the fields of self-developing photography, dosimetry, optical signal processing, data display, decoration,
10 control of radiation intensity and others. The most widely used and commercially successful photochromic material technology is in eyewear for protecting against sunlight and controlling of sunlight intensity. Currently, the majority of the photochromic eyewear lenses are made of
15 silver halide based glasses. However, photochromic plastic lenses based on spirooxazine photochromic compounds have made some inroad.

The spirooxazine compounds are a member of a family of photochromic compounds with inherently excellent photo-
20 durability. They are the preferred photochromic compounds used for producing eyewear glasses (both ophthalmic and sunglass lenses). This is due to their photo-fatigue resistance and their colourless hue in the unactivated form (i.e., in the absence of the activating ultraviolet light).

25 Many methods are known to impart to the photochromic property to a material. The commonly used methods include dyeing, casting, coating and injection molding. The method of application depends strongly on the substrate material. For example, the spirooxazine dyes are sensitive to certain
30 chemicals such as polymerization initiators and oxidants, and are unstable at high temperatures (e.g., at injection molding temperatures).

The commonly used plastics for eyewear included poly(methyl methacrylate) (PMMA), cellulose acetate
35 butyrate (CAB), polycarbonate, and cellulose acetate propionate (CAP) and diethylene glycol bis (allyl

carbonate) (CR-39). The first four are thermoplastics while CR-39 is a thermoset plastic. Since the methods for photochromic treatment depend on the nature of the lens substrate, some methods applicable to the thermoplastics may not be useful for the thermoset plastics. For example, the photochromic dye cannot be mixed into CR-39 monomer to be polymerized along with the monomers to obtain a product with the dye uniformly dispersed, since the polymerization catalyst will destroy the dye.

10 The dyeing process is generally performed in a high boiling organic solvent bath. The solvents commonly used are alcohols, glycols, aromatic or aliphatic hydrocarbons. The thermoset plastic lens is immersed in the solvent bath which normally contains between 1% to 10% photochromic dye. 15 The dye bath is maintained in the range of 90° to 120°C and the immersion time is normally one to several hours. This will ensure that an adequate amount of dye is diffused into the lens surface for sufficient photochromic durability. Thermal stability of the dye in the dyeing bath is a major 20 problem because of the cost associated with high dye concentration and long dyeing time.

Two other known processes for application of photochromic dye to the surfaces of a lens include vapor-liquid phase transfer and solid phase transfer. In the 25 vapor-liquid phase transfer, the lens is heated in the presence of vapours of photochromic dye. In the solid phase transfer, the photochromic dye and a film-forming resin are dissolved in a solvent. The resulting solution is applied to the surface of a lens by any of the commonly used 30 techniques such as dipping, spinning or spraying. The coated lens is heated to a high temperature below the melting point of the dye for a sufficient length of time to allow diffusion of the dye from the resin film to the lens substrate. Generally, the heating temperature is in the 35 range of 110° to 150°C from several minutes to hours depending on the hardness of the lens. After the heat

treatment step, the resin-film is removed. The heating source for these two photochromic processes is a conventional heat oven.

Heating by conventional ovens is convenient, but not cost effective, especially for hard cured lenses. To obtain adequate amounts of dye transferred to the lens, several hours of heating time is required.

The object of the present invention is to propose a more efficient method for producing photochromic plastic lenses. The object is achieved by the method for producing photochromic plastic lenses of the present invention as claimed in claim 1. In accordance with the present invention, a photochromic property is imparted to a plastic lens (e.g., by dyeing the surface of the lens with a photochromic dye solution) at high temperature by microwave heating for a predetermined amount of time.

The microwave heating, in accordance with the present invention, considerably reduces the heating time as compared to the convection heating used by the prior art. Accordingly, the microwave heating results in an efficient, cost effective method for producing photochromic plastic lenses.

The above-discussed and other features and advantages of the present invention will be appreciated and understood by those skilled in the art from the following detailed description.

A novel method for producing a photochromic plastic lens is presented. The plastic lens is comprised of any optical grade plastic material, such as poly(methyl methacrylate) (PMMA), cellulose acetate butyrate (CAB), polycarbonate, cellulose acetate propionate (CAP) or diethylene glycol bis(allyl carbonate) (CR-39). The first four are thermoplastics while CR-39 is a thermoset plastic. Other plastic materials having a refractive index greater than CR-39 have been developed for eyewear application. For

example, CR-400s materials from PPG and MR-6 from Mitsui Toatsu.

As was noted in the prior art, spirooxazine photochromic compounds are being used for photochromic plastic lenses and such is contemplated by the present invention. The spirooxazine compounds are a member of a family of photochromic compounds with inherently excellent photo-durability. They are the preferred photochromic compounds used for producing eyewear glasses (both ophthalmic and sunglass lenses). This is due to their photo-fatigue resistance and their colourless hue in the unactivated form (i.e., in the absence of the activating ultraviolet light). Representative spirooxazine family of photochromic compounds can be classified into several subclasses, which are spiroindolinonaphthoxazines (NISO), spiroindolinopyridobenzoxazines (QISO), spiroindolinobenzoxazines (BISO) and spiroindolinoanthryloxazines (AISO).

In accordance with the present invention a photochromic property is imparted to the lens by dyeing the lens in photochromic dye solution. The lens is immersed in the photochromic dye solution which is then heated for a predetermined amount of time. After heating the lens may remain in the heated photochromic dye solution to seek. The lens is then removed and washed with a solvent.

The heat treatment step is accomplished by microwave heating. The microwave heating has been found to be very efficient and cost effective as compared to the prior art method. The heat treatment step in the prior art was accomplished by conventional heating. Heat treatment using the conventional ovens required a heating temperature in the range of 110° to 150°C (for CR-39 lenses) for several minutes to hours depending on the hardness of the lens. Heating by a conventional oven is convenient, but not cost effective especially for hard high cured lenses. To obtain an adequate amount of dye transferred to the lens, several

hours of heating time is required. Microwave heating, in accordance with the present invention, considerably reduces the heating time as can be seen from the examples provided below.

5 The lenses used in the below examples were high cured AOLite ^(RTM) CR-39 plano lenses having a diameter of 76 mm (manufactured by American Optical Corporation, the assignee of the present invention). The photochromic dye used was a mixture of 1,3,3,4,5- and 1,3,3,5,6-pentamethyl-9'-methoxy
10 NISO isomer mixture. Further, in order to determine the amount of dye being transferred to the lens substrate, the absorbencies of the lens at 360 nm was measured before and after photochromic treatment.

Example 1

15 In a first prior art example, 1.2 kg of propylene glycol was added in a 1.5 L glass beaker. The solvent was heated to 115°C with a hot plate. Twenty-four (24) g of photochromic dye were added to the solvent. Two AOLite ^(RTM) lenses were used, one lens was immersed in the dye bath for
20 1 hour and the other 2 hours. The absorbence change at 360 nm before and after treatment for the first lens (i.e., 1 hour treatment) was 0.18 and for the second lens (i.e., 2 hour treatment) was 0.25.

Example 2

25 In a second prior art example, a 2% dye bath solution was made in a metal pan and heated to 152°C by a hot plate. Two AOLite ^(RTM) lenses were dyed by immersing them in the dye bath for 1 hour. The dye uptake as measured by the absorbence change at 360 nm was 0.46 for both lenses.

Example 3

30 In a first example in accordance with the present invention, a 1 L glass beaker containing 500 g propylene glycol and 10 g photochromic dye was placed in a microwave oven (e.g., Goldstar ^(RTM) 800 watt model) with a lens therein
35 and heated for 6 minutes. The lens was then soaked in the heated solution for 15 minutes. The lens was then removed

and washed with acetone. The temperature of the solution after heating was 167°C. The absorbance change of the lens at 360 nm was 0.37.

Example 4

5 In a second example in accordance with the present invention, 5 g more dye was added to the solution of Example 3 to make a 3% dye solution. A lens was dyed by immersing it in the solution and heating the solution with the lens therein for 6 minutes, using the microwave oven.
10 The lens was then removed and washed with acetone. The absorbance change of the lens at 360 nm was 0.60.

The method of the present invention obtains comparable results to that of the prior art in significantly less time. This is clearly seen by comparing the time requires
15 in the prior art examples 1 and 2 to examples 3 and 4 which are in accordance with the present invention.

While the preferred method of imparting the photochromic property to the lens is dyeing, other known methods, such as casting, coating and injection molding may
20 be employed. The method of application is dependent on the lens material used. For example, spirooxazine dyes are sensitive to certain chemicals, such as polymerization initiators and oxidants, and are unstable at high temperatures (e.g., at injection molding temperatures).
25 Further, some methods applicable to the thermoplastics may not be useful for the thermoset plastics. For example, the photochromic dye cannot be mixed into CR-39 monomer to be polymerized along with the monomers to obtain a product with the dye uniformly dispersed since the polymerization
30 catalyst will destroy the dye.

Alternately, the aforementioned solid phase transfer process for applying the photochromic dye to the surface of the lens may be used in the method of the present invention. In the solid phase transfer, the photochromic
35 dye and a film-forming resin are dissolved in a solvent. The resulting solution is applied to the surface of a lens

by any of the commonly used techniques such as dipping, spinning or spraying. The coated lens is then heated to a high temperature below the melting point of the dye for a sufficient length of time to allow the diffusion of the dye
5 from the resin film to the lens substrate.

While preferred embodiments have been shown and described, various modifications and substitutions may be made thereto without departing from the spirit and scope of the invention has been described by way of illustrations
10 and not limitation.

CLAIMS

1. A process for producing a photochromic plastic lens,
including the steps of:
mixing a photochromic dye solution;
5 immersing a plastic lens in said photochromic dye
solution; characterised by
microwave heating the photochromic dye solution with
the plastic lens therein for a predetermined amount of
time;
10 removing the plastic lens from the heated photochromic
dye solution after a predetermined amount of time has
elapsed; and washing the plastic lens with a solvent.
2. The process of claim 1, characterised in that said
photochromic dye solution comprises a spirooxazine
15 compound.
3. The process of claim 2, characterised in that said
spirooxazine compound comprises spiroindolino-
naphthoxazines, spiroindolinopyridobenzoxazines,
spiroindolino-benzoxazines or spiroindolino-
20 anthryloxazines.
4. The process of any of the claims 1 to 3,
characterised in that said step of microwave heating
comprises microwave heating using a microwave oven.
5. The process of any of the claims 1 to 4,
25 characterised in that said plastic lens comprises a
thermoplastic or a thermoset plastic.
6. The process of any of the claims 1 to 5,
characterised in that said plastic lens comprises
poly(methyl methacrylate) (PMMA), cellulose acetate
30 butyrate (CAB), polycarbonate, cellulose acetate propionate
(CAP) or diethylene glycol bis(allyl carbonate) (CR-39).
7. The process of any of the claims 1 to 6,
characterised in that said photochromic dye solution
includes from about 1% to about 10% of a photochromic dye.
- 35 8. A process of any of the claims 1 to 7, characterised
by the additional steps of soaking the plastic lens in the

microwave heated photochromic dye for a predetermined amount of time and removing the plastic lens from the heated photochromic dye solution after the predetermined amount of time for soaking has elapsed.

- 5 9. A process for producing a photochromic plastic lens, characterised by the steps of:

coating the surface of a plastic lens with a photochromic dye solution;

- microwave heating the coated plastic lens for a
10 predetermined amount of a time, whereby a photochromic dye is diffused from a resin film to the surface of the plastic lens; and

removing the resin film.

10. The process of claim 9, characterised in that said
15 step of coating comprises dipping the lens in the photochromic dye solution.

11. The process of claim 9, characterised in that said step of coating comprises spin coating the surface of the plastic lens with the photochromic dye solution.

- 20 12. The process of claim 9, characterised in that said step of coating comprises spraying the surface of the plastic lens with the photochromic dye solution.

13. The process of any of the claims 9 to 12, characterized in that said plastic lens comprises a
25 thermoplastic or a thermoset plastic.

14. The process of any of the claims 9 to 13, characterised in that said plastic lens comprises poly(methyl methacrylate) (PMMA), cellulose acetate butyrate (CAB), polycarbonate, cellulose acetate propionate
30 (CAP) or diethylene glycol bis(allyl carbonate) (CR-39).

15. The process of any of the claims 9 to 14, characterised in that said photochromic dye solution comprises said photochromic dye and said resin dissolved in a solvent.

- 35 16. The process of claim 15, characterised in that said photochromic dye comprises a spirooxazine compound.

17. The process of claim 16, characterised in that said spirooxazine compound comprises spiroindolino-naphthoxazines, spiroindolinopyridobenzoxaines, spiro-indolinobenzoxazines or spiroindolinoanthryloxazines.

5 18. The process of claim 15, characterised in that said solvent comprises an alcohol, a glycol, an aromatic hydrocarbon or an aliphatic hydrocarbon.

19. The process of any of the claims 9 to 18, characterised in that said step of microwave heating
10 comprises microwave heating using a microwave oven.

20. The process of any of the claims 9 to 19, characterised in said step of removing comprises removing the resin film using a solvent.

21. A process substantially as hereinbefore described.

Patents Act 1977
Examiner's report to the Comptroller under
Section 17 (The Search Report)

- II -

Application number

GB 9315620.6

Relevant Technical fields

(i) UK Cl (Edition L) G2J (JFP, JS3A, JS3X, JS6C2,
JS6CX, JS6DX)

(ii) Int Cl (Edition 5) G02C

Search Examiner

R E HARDY

Databases (see over)

(i) UK Patent Office

(ii) WPI

Date of Search

7 SEPTEMBER 1993

Documents considered relevant following a search in respect of claims ALL

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
A	GB 2174711 A (PPG)	9
A	US 4289497 A (HOVEY)	1

SF2(p)

SJ - doc99\fil001152

Category	Identity of document and relevant passages - 12 -	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

This Page Blank (uspto)